PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald R. Huffman, et al. **Examiner:** P. DiMauro

Serial No.: 08/486,669 **Art Unit:** 1754

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For: NEW FORM OF CARBON

Assistant Commissioner for Patents
Washington, DC 20231

SUPPLEMENTAL DECLARATION OF DONALD R. HUFFMAN UNDER 37 C.F.R. §1.131

Sir:

I, Donald R. Huffman, declare and say as follows:

1. I am a co-applicant of the above-identified application.

2. The other co-inventor of the above-identified application is Wolfgang Kratschmer, with whom I have collaborated. Although Dr. Kratschmer conducted his research at the relevant time at the Max Planck Institute in Germany, during the course of our collaboration, we have regularly communicated with one another, exchanging ideas, concepts and experimental details and results. In addition, we have visited each other's laboratories and have conducted additional research therein during our visits relating to the subject matter of the present invention described in the above-identified application. All of our combined activities have led to the completion of the invention described and claimed in the above-identified application.

3. I am currently a Regent's Professor of Physics, at the University of Arizona. I have received several accolades and awards relating to the subject invention, which include, inter alia, a Material Research Society Annual Medal Award in 1993, which I shared with Dr. Kratschmer, for the "Discovery of a Way to Produce Macroscopic Quantities of the Fullerenes and for Ellucidating (sic) Many of the Physical and Chemical Properties", and the Hewlett-Packard EuroPhysics Prize in 1994, which I shared with Drs. Kratschmer, Smalley and Kroto, for the "Discovery of New Molecular Forms of Carbon and their Production in the Solid State".

My curriculum vitae which lists, inter alia, my awards and honors and publications, is attached hereto as Exhibit A. (Exhs. A-1 to A-8).

4. It is my understanding that the United States Patent and Trademark Office cited a paper by Kratschmer, et al. published in Chemical Physics Letters, 1990, 167-170 ("Kratschmer, et al.") in support of a rejection of the above-identified application.

5. It is my understanding that Kratschmer, et al. published on July 6, 1990.

6. The invention described and claimed in the above-identified application was completed in the United States prior to July 6, 1990, i.e., the publication date of Kratschmer, et al.

7. The present invention is directed to a method of producing fullerene-60 and fullerene-70 as species of fullerenes in macroscopic amounts. An integral part of the present invention comprises vaporizing elemental carbon, e.g., graphite, in the presence of an inert quenching gas under conditions effective to form a soot comprising fullerenes, e.g., fullerene-60, which species of fullerenes are present in the sooty carbon product in macroscopic amounts. Proving that macroscopic amounts of fullerene species, e.g., fullerene-60, are present in the soot required isolation of the same from the soot. Thus, in addition to the step of producing species of fullerenes, e.g. fullerene-60, in macroscopic amounts, much of the activity described hereinbelow focused on proving that the species were produced in macroscopic amounts . Thus, we undertook to isolate fullerene-60 and fullerene-70, as species of fullerenes, from the soot.

8. As evidence that these acts, including the completion of the present invention in the U.S., occurred prior to the publication of Kratschmer et al., annexed hereto and made a part hereof are Exhibits B-I consisting of photocopies of laboratory records of experiments conducted in the laboratories at the University of Arizona.

9. The acts reported in the laboratory notebook entries were conducted prior to July 6, 1990, the publication date of Kratschmer, et al. either by myself or by someone working under my direction and control.

10. Data not pertinent to this invention and dates have been masked out in the preparation of these photocopies.

11. To enhance the understanding of the present process as to the acts described herein, reference is made to Exhibit B, which is a photocopy of 4 pages from Dr. Lowell Lamb's laboratory notebook, identified as Pages B-1 to B-4. Dr. Lamb, at the relevant time, was a graduate student working in my laboratory under my supervision and control.

12. Exhibit B summarizes in detail an embodiment of the present invention for producing fullerene species, e.g., fullerene-60, in macroscopic amounts. It describes that graphite rods are vaporized in an inert atmosphere of helium, e.g., 100 torr of helium, in a belljar apparatus. Above the rods is a chimney made out of a 2" diameter quartz tube topped with two microscopic slides to collect the vaporized carbon smoke. The carbon smoke is scraped off the chimney and sides of the chamber, and placed in benzene. The benzene is evaporated off until a brownish gold residue remains, then the brownish gold residue is sublimed in an atmosphere of inert gas such as helium. The sublimed material is collected on a quartz substrate. In each of the instances wherein the product was isolated, it was produced in amounts that could be seen with the naked eye.

13. One product of the procedure described hereinabove in paragraph 12 is a relatively pure fullerene-60 molecule in macroscopic amounts. This is verified by the UV-

VIS spectrum, in which one observes a camel structure in the absorption pattern, e.g., three specific absorptions at about 220, 270 and 340nm in the UV. Since the absorption between 240 and 270 nm reminded us (Kratschmer and myself) of camel humps, we designated the spectra as camel humps. (The three absorptions turned out to be associated with and is reflective of the presence of fullerene-60 and fullerene-70 in the sample).

14. An example of such a spectrum is depicted on pages B-3 and B-4, which are photocopies of additional pages in Lowell Lamb's notebook. Although the spectra are in color in the notebooks, the colors did not reproduce in the original photocopying. I have therefore retraced the lines with the appropriate colors on these pages of the exhibit and have written the appropriate color designations above and/or below the lines.

15. In the experiments described hereinbelow, the sooty carbon product was obtained by following the procedure outlined hereinabove. The emphasis in these experiments was to definitely prove that macroscopic amounts of fullerene species, e.g., fullerene-60, were produced. Thus, the emphasis in many of the exhibits is to separate the product produced in accordance with the procedure described herein from the soot and to show by measuring physical characteristics, such as UV spectra, IR spectra, X-ray diffraction pattern, and the like that the present process produced species of fullerenes, e.g.,

fullerene-60 and that they were produced in macroscopic amounts.

16. In the experiment described on pages B-3 and B-4, Lamb had followed the procedure described hereinabove and prepared fullerene species, e.g., fullerene-60, from soot, as described in paragraph 12. He had separated the fullerene products from the carbon sooty product by sublimation. More specifically, he had sublimed the mixed fullerene products, containing, among other things, fullerene-60 and fullerene-70, from the soot, prepared in accordance with the procedure described in Paragraph 12 herein in a helium atmosphere until a thin film was formed on the surface of the quartz substrate. According to the procedure described therein, he removed the film from the quartz substrate and took the UV spectra of the collected material. As outlined in the notebook he continued subliming the material in the soot until another film appeared, which, he again isolated and scanned. He repeated this process until no more material was collected on the quartz substrate. It is noted that in the spectrum located on the right side of Page B-3, there are blue and red lines which show absorption at about 230, 270 and 340 nm. These absorptions turned out to be associated with and reflective of the presence of fullerene-60 and fullerene-70 in the sample. This again is illustrated by the blue and the red lines in the spectra located on the left side on page B-4.

17. Exhibit C is a photocopy of 9 pages of my notebook, identified as C-1 to C-9. These pages describe the vaporization of carbon in an inert atmosphere to form the carbon sooty product, as described herein, the isolation of the carbon soot and separation by sublimation of the fullerenes, e.g., fullerene-60.

18. Prior to any sublimation, I took the UV of the sample of carbon soot produced and isolated from the sides of the chamber in accordance with the procedure described herein. The UV confirmed the presence of fullerene species, e.g., fullerene-60, in the soot.

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19. Pages C-1 to C-5 describe various separations of fullerene-60 from the collected soot by sublimation. Attention is directed to Pages C-4 and C-5, which not only describes a sublimation of the fullerene-60 from the soot, but also provides the spectra showing the camel humps referred to hereinabove, respectively. This spectra clearly evidence that the product contained fullerene species, e.g., fullerene-60.

20. Pages C6 and C7 describe additional sublimation experiments that were used to separate the fullerene-60 produced in macroscopic amounts from the soot. In the experiments described therein, a 1cm x 2cm microscope slide which had been heavily coated with carbon soot in accordance with the procedure described in paragraph 12 hereinabove, was heated. The heating was effected in a small quartz crucible surrounded by tungsten wire in the bell jar filled with about

one atmosphere of helium. The quartz substrate was placed just above the crucible for collecting the sublimed material. To prove that I had prepared the fullerene-60 in macroscopic amounts, I performed several sublimations and scanned the sublimed product each time. A typical UV is provided on Page C-9.

21. The UV spectra on page C-9 clearly shows the presence of the camel humps, and this clearly indicates that fullerene-60 was produced by the process described hereinabove.

22. Exhibit D is a photocopy of two pages of a laboratory notebook of Lowell Lamb. The sooty carbon product comprising macroscopic amounts of fullerene-60 was prepared as above. The isolation of a fullerene species, e.g., fullerene-60, from carbon soot and the purification of same, was effected by sublimation. Attached to the bottom of Page D-1 and Page D-2 is the UV and visible spectra, respectively, of the fullerene-60 product so obtained.

23. On the graph on the bottom of Page D-1, attention is drawn to the UV absorptions at 240, 270 and 340nm again indicating the presence of fullerene-60 in the sample.

24. Exhibit E is a photocopy of three pages of Lowell Lamb's laboratory notebook. Page E-1 is a visible spectra of fullerene-60, prepared in accordance with the procedure described hereinabove and shows absorption at about 415, 500, and 670nm, which is indicative of fullerene-60.

Page E-2 describes modifications of the procedure described on Page 92 and 93 of the notebook (Pages B-1 and B-2). Moreover, it refers to an IR spectrum of fullerene-60 on NaCl produced in accordance with the procedure outlined on Pages B-1 and B-2. It refers to the absorption of the fullerene-60 at 1410 and 1180 cm., which turns out to be associated and reflective of the presence of fullerene-60. Page E-3 is a copy of IR spectra of fullerene-60 on NaCl referred to on Page E-2.

25. Exhibit F is a photocopy of relevant portions of a progress report which was written in Lowell Lamb's laboratory notebook. Page F-3 comments on the IR and UV spectra of the fullerene-60 sample obtained and reports that the procedure described in Exhibit B produces a fullerene-60 product in approximately 0.1 gram batches.

26. The fullerene products, produced in accordance with the procedure described hereinabove, were soluble in non-polar solvents and insoluble in polar solvents. This is indicated in Exhibit G, which is a photocopy of two pages of my notebook.

27. Exhibit G consists of two pages, Page G-1 and G-2. Page G-1 describes the tests which I conducted regarding determining the solubility of the fullerene product. I found that it is soluble in benzene, CS_2 and CCl_4, but insoluble in water, acetone, methanol and proponal.

28. The fact that the fullerene product is found to be soluble in non-polar solvents, while the soot was insoluble in the non-polar solvent was evidence that non-polar solvents could be used to extract the fullerene product from the soot. Thus, this represented an alternate means for separation of the fullerene product from the soot.

29. Page G-2 is the UV/VIS spectrum of the fullerene product dissolved in benzene.

30. The spectra referred to in paragraphs 21 and 29 are of exceptional quality and clearly show the presence of fullerene-60.

31. Exhibit H consists of one page and is an X-ray diffraction of the fullerene powder produced in accordance with the procedure described hereinabove. The spectrum is identical to the ones we and others published with respect to fullerene-60.

32. Exhibit I, consisting of one page, is a mass spectrum of the fullerene material produced in accordance with the procedure described hereinabove. It clearly shows the presence of two species of fullerenes, e.g., fullerene-60 (mass 720) and fullerene-70 (mass 840) in a single ionization, along with some breakup products of fullerene-60 such as doubly ionized fullerene-60.

33. These exhibits demonstrate that a process for the preparation and isolation of various fullerene species, e.g., fullerene-60 and fullerene-70 as species of fullerenes,

in macroscopic amounts has been performed by myself or under my direct supervision and control in the United States prior to the publication date of Kratschmer et al.

34. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: May 10, 2000

Donald R. Huffman
DONALD R. HUFFMAN

MJC:ahs/bb

Curriculum Vitae

Donald R. Huffman
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DATE AND PLACE OF BIRTH: June 19, 1935: Fort Worth, Texas

EDUCATION:

1957 B.S. (Physics) Texas A&M University
1959 M.A.(Physics) Rice University
1966 Ph.D.(Physics) University of California, Riverside
1967 NSF Postdoctoral Fellow, University of Frankfurt, Germany

POSITIONS HELD:

1959-60 (and summers of 1960, 1961 and 1962) Research Engineer, Production Research Division of Humble Oil Company, Houston, Texas
1960-62 Instructor in Mathematics and Physics, Pepperdine University
1968-70 Assistant Professor of Physics, University of Arizona
1970-75 Associate Professor of Physics, University of Arizona
1975-76 (summers) Visiting Scholar, Department of Applied Mathematics and Astronomy, University College, Cardiff, Wales
1975-76 (Sabbatical leave) Visiting Scientist, Max-Planck Institute, Stuttgart; European Space Agency, Noordwijk, Holland.
1983-84 Humboldt Senior US Scientist Awardee; visiting scientist at Max-Planck Institutes for Nuclear Physics (Heidelberg) and Solid State (Stuttgart)
1975-93 Professor of Physics, University of Arizona
1993- Regents' Professor of Physics, University of Arizona
1993 (fall semester sabbatical) Visiting Scientist, Max Planck Institute for Nuclear Physics, Heidelberg

AWARDS and HONORS:

1982-83 Alexander von Humboldt, Senior US Scientist Award
1993 Regents' Professor , University of Arizona
1993 Materials Research Society Annual Medal Award (w/ W. Krätschmer)
"For the Discovery of a Way to Produce Macroscopic Quantities of the Fullerenes and for Ellucidating Many of their Physical and Chemical Properties"

1994 Hewlett-Packard Europhysics Prize
(with W. Krätschmer, R. Smalley and H. Kroto)
for "Discovery of New Molecular Forms of Carbon and their Production in the Solid State"

1994 Distinguished Alumni, University of California, Riverside
1994 Doctor of Laws (honorary) Pepperdine University
1998 Distinguished Alumnus Award, College of Science, Texas A&M

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1968-70 Assistant Professor of Physics, University of Arizona
1970-75 Associate Professor of Physics, University of Arizona
1975-76 (summers) Visiting Scholar, Department of Applied Mathematics and Astronomy, University College, Cardiff, Wales
1975-76 (Sabbatical leave) Visiting Scientist, Max-Planck Institute, Stuttgart; European Space Agency, Noordwijk, Holland.
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1998 Distinguished Alumnus Award, College of Science, Texas A&M

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PATENTS

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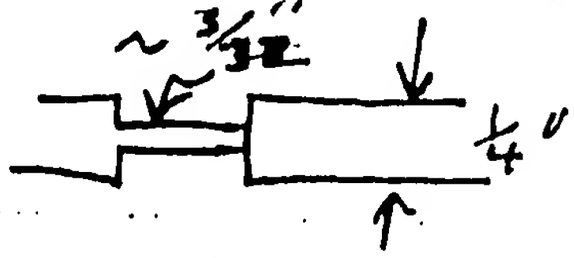
B

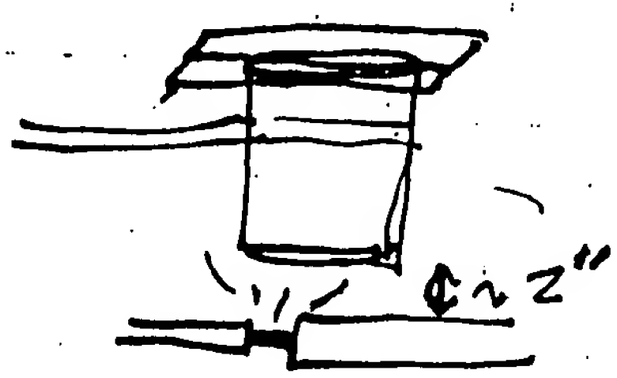
Journal Entry

In the last three days, I've made some progress in the production of C_{60}. Below is the process.

① Evaporate Carbon in ~ 100 Torr of He.* The tip dimensions are



Above the rods I have a "chimney" made out of a ~ 2" diameter quartz tube topped with two microscope slides to collect the smoke



(*Flush 3 times with ~ 100 Torr of He)
↑ First

Set the variac to ~ 75; If the tip breaks and two thin sections connect the ¼" rods, turn the voltage to 85.

② Repeat ~ 5 times.

③ Scrape smoke off Chimney + Sides of chamber.

④ Combine smoke + benzene in a testtube. Stir thoroughly.

⑤ Ultrasonic for ~ 2 min.

⑥ Centrifuge on 4 for ~ 10 min.

⑦ Pour Dark red liquid into quartz crucible - place crucible on warm hot plate (cool enough to be touched for a few seconds) and heat until all benzene has evaporated. A brownish golden residue should remain.

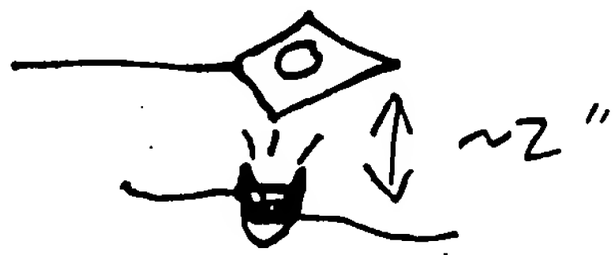
READ BY John J. Emmert

Pg 2 →

EXH. B-1

(8) Put crucible with residue into wire basket in vacuum chamber.

(9) Flush 3 times with 100 Torr of He – At 100 Torr of He, sublime C_{60} onto quartz substrate at ~30 V on Variac until film appears on substrate.



(10) Remove substrate + scan from 400–200 nm.

(11) Clean substrate and Repeat (9) & (10) until all of the offensive volatiles have been driven off. This will have happened when the spectrum resembles the brown spectrum taped in on page 94. The blue and purple spectra are of samples which still contain the unknown volatile. What remains in the crucible is C_{60}.

Temperature Dependence

Four Taped in on page 95 are ~~three~~ scans of a C_{60} sample

(1) Blue – Room Temperature
(2) Green – Immediately after immersion & equilibration in liquid N_2.
(3) Purple – 2nd N_2 Temp scan
(4) Red – Final Room Temp scan.

Broad feature (features?) in visible around 425–525 nm appears real.

READ BY JOHN T. EMMERT

John T. E

Spectra of C_{60} at Various stages of Volatiles removal

0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1

1.0

Abs = 2.0

Visible

Red
Blue
Purple
Brown

Blue - 1st Coating
Purple - 2nd "
Brown - 3rd "
Red - 4th " (heavy)

EXH. B-3

REDACTED

0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0

Green
Green
Red
Blue
Red
Green
Green

1.0 Abs

1.0 Abs

0.5 Abs

Red
Red
Blue

UV

Visible

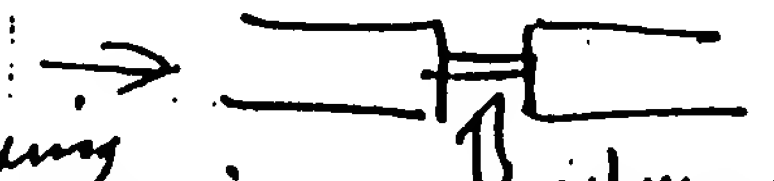
(1) Blue – Room Temp
(2) Green – Dipped in Liquid N_2
(3) Red – Room Temp

A (nm)

C_{60} making

REDACTED

10:40 AM - Try for ~100 torr ≈ 380 torr on gauge according to calibration in Lowell's notebook.



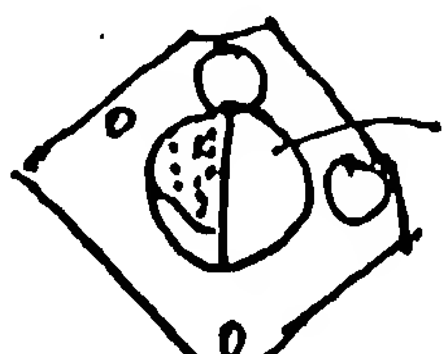
#1 UV spectrum on Cary 118 shows a good C_{60} spectrum just as in our paper.
Peak OD = ~1.4.
Scraped off with razor blade onto glassine paper.

#2 UV spectrum shows similar result.
Peak OD ≅ 2.25. On this production I [illegible] the rods to allow the usual residue of low temperature stuff to come off & disperse

Used samples #1 & #2 scraped off to try to sublimate C_{60}.

Multistrand tungsten wire coil bent crudely into a shape that would hold a clear quartz crucible.
Heated to about 600°C in vacuum & then something great:

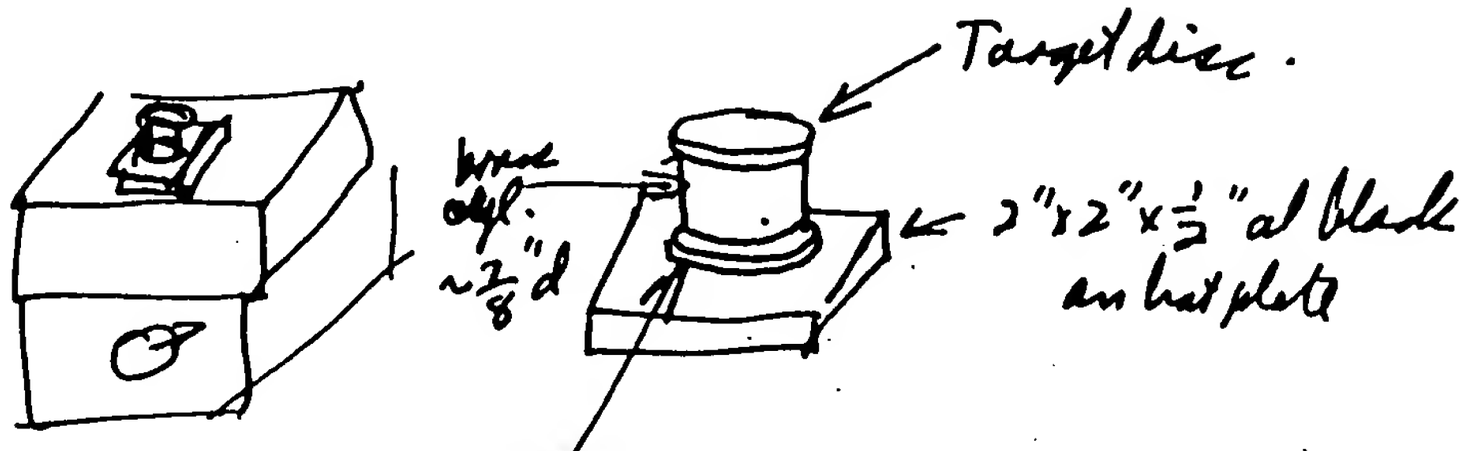
UV spectrum is inconclusive until better background is seen:



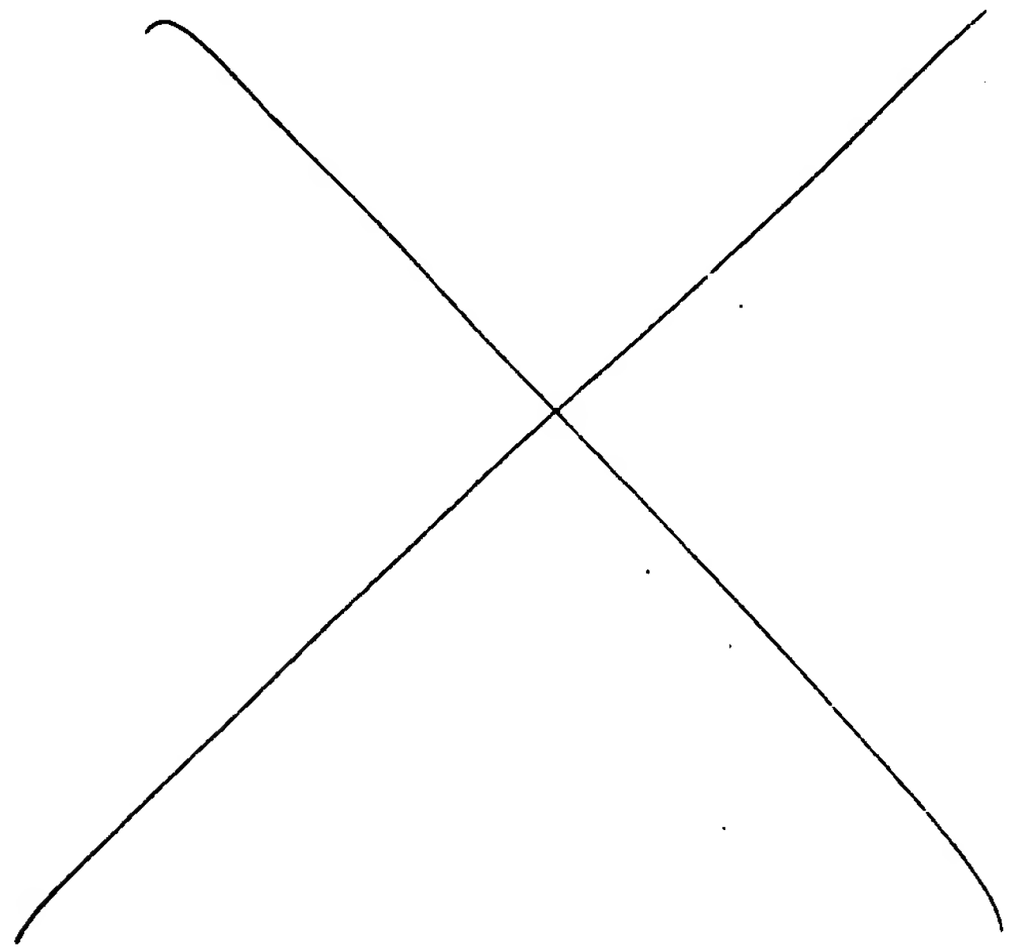
(cont.)

(cont.)

Attempt at Sublimation of C_{60} in Air on Hot Plate.



Heated for 10 min in the above arrangement. No indication of anything on target disc. Re-analysis of UV spectrum of source disc shows change over from $\rightarrow$ Cusp structure.



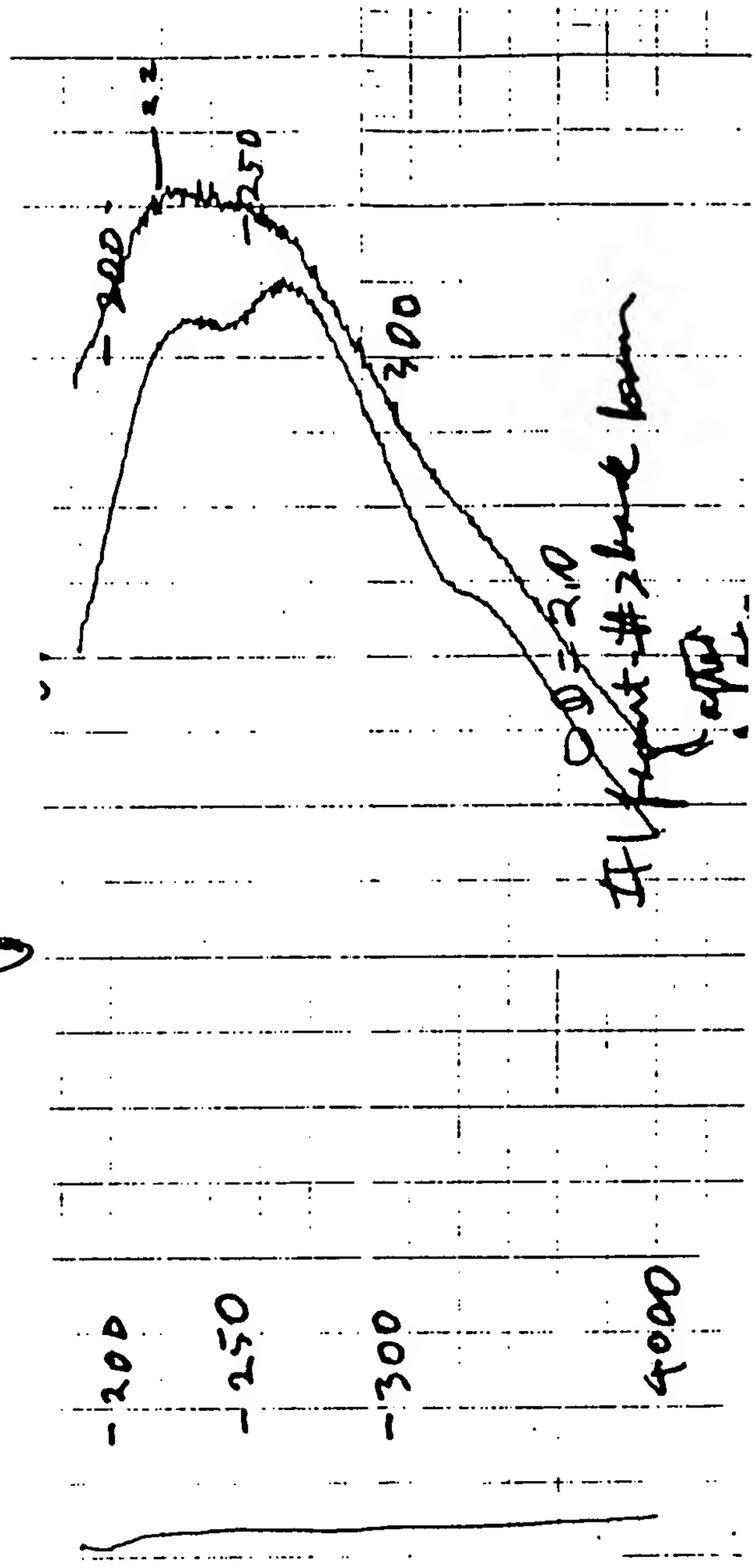
EXH. C-2

See next page

REDACTED

75

(cont.)

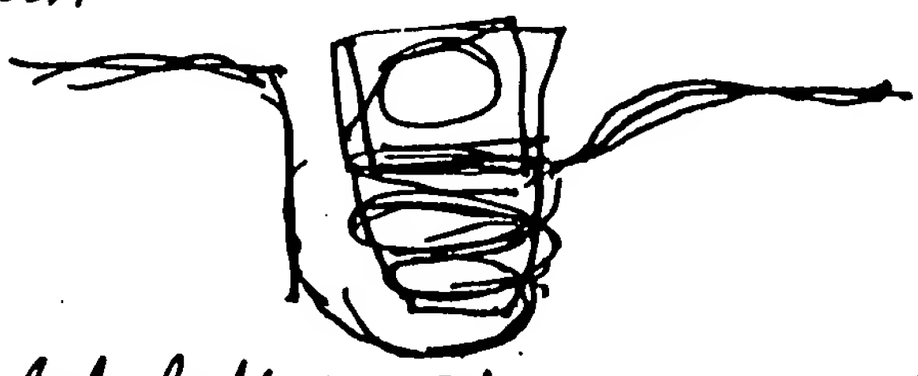


EXH. C-3

76

(cont.)

Fri night top of crucible C60 I wound a new coil around the clean quartz crucible and tip 3 stranded tungsten.



Placed ab. holder with silica disc just above the crucible.
Note: perspective of drawing above is not good.
Scraped carbon off the sample holder from a previous run -- also collected some by scraping from posts and other hardware in the chamber.

Flushed chamber w/ He & filled to ~ $\frac{3}{4}$ atm. Heated filament ~ 15 amp until I observed something on disc.

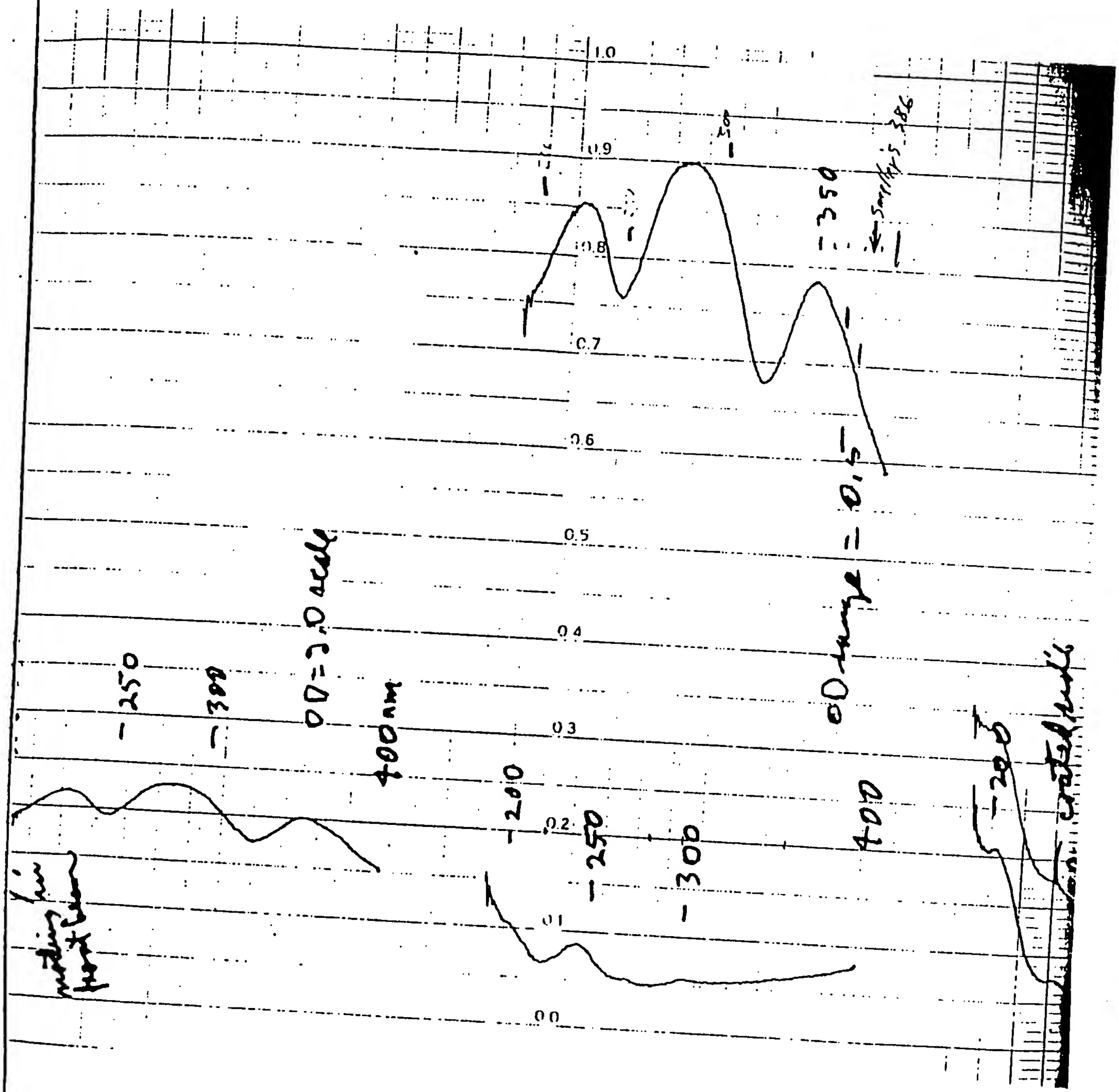
Spectrum opposite page → shows that I indeed succeeded in concentrating C_{60}.

The sample again appeared bluish by scattered light in the forward direction and perhaps reddish by transmission. Most white areas flakes of the fluffy carbon that seems to have covered the surface.

I will try to get them this by not scraping off the carbon into fluffy aggregates.

REDACTED

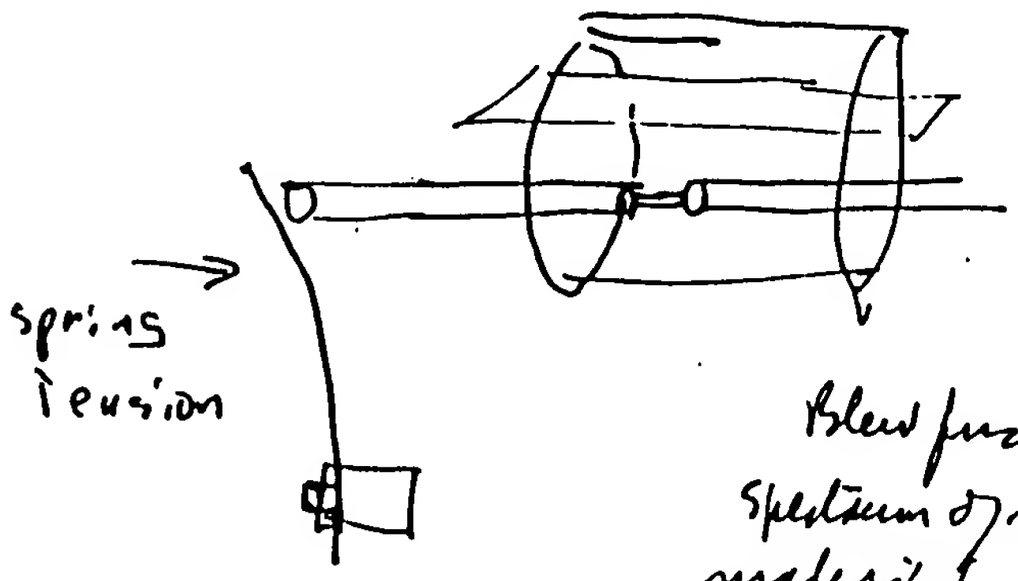
77



EXH. C-5

Made C_{60} making

#1 Tried 3mm diameter rod in qtz. tube w/ microscope glass as drawn below:



3mm is
Blew fuse - too large.
Specimen of soot in tube shows molecule C_{60} signature.

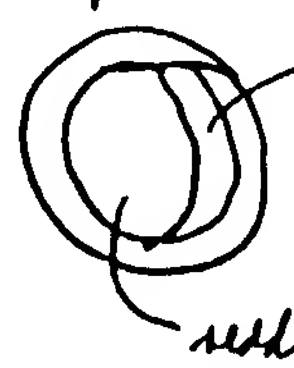
#2 Trial
Same as above but 1-2 mm dia. rod, a little longer than used (~1cm). Rod snapped when heating started but smoke continues as shown here part was pushed by spring expanded stock.
Heavy coating on inside of pyrex tube slide.
Will try again with a 1/2mm x ~8 mm tip to get more C_{60}. Then try to concentrate it.

#3 Trial
Heated rather quickly. Tip collapsed. ~5 sec in all today to prevent drawing off the C_{60}.

#4 Microscope slide from #2-3 with heavy coating is broken into a few pieces to fit into crucible. Idea is to try to present the fluffy contaminant from yesterday by sublimin directly from coated slide.

EXH. C-6

cont.
Heated filament to ~ 10 amp on meter. Filament misbehaving
Heating more towards electrode. Later settled down so that uniform
red heating occurred. 40 on variac - 20 on current meter.
Left it for ~ 3 min while I observed deposition using
microscope light from above.



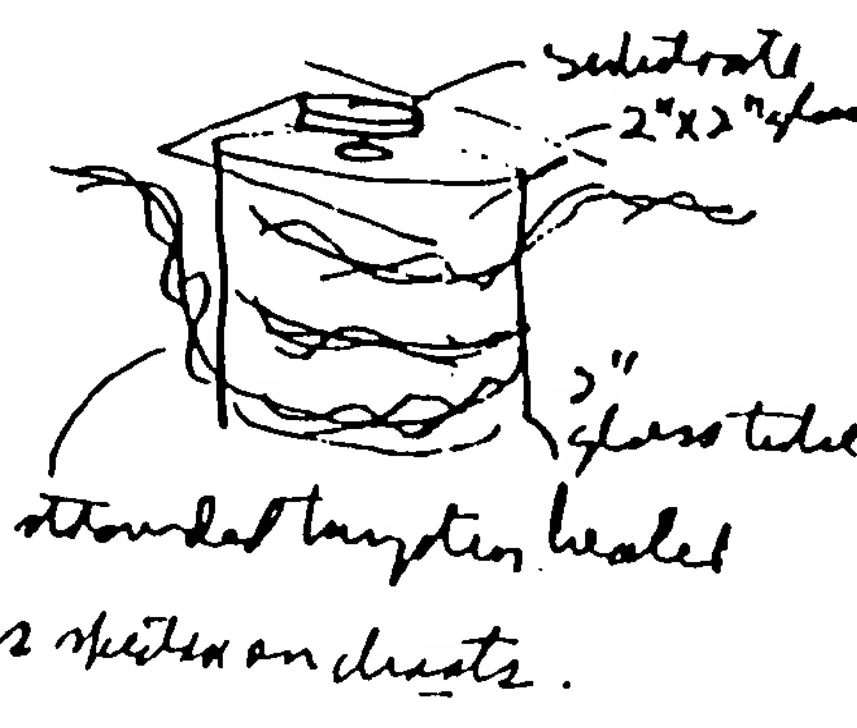
Two spots resulted. Ran spectrum of each. Totally different
Don't understand the blue-by-trans. part.

Cleaned substrate - will now try same charge as drawn on (bottom) to see if any C_{60} is left.

Cont. 3 min - 30 v - 12.5 amp on primary
appears to be same sort of smoke cloud present & visible in chamber as viewed by microscope lamp into chamber in darkened room. No evidence of any more C_{60} or anything else depositing on substrate.

Next try an uncoated portion (~1cm x 1cm) of heavily coated microscope slide (#1 & #2). Increased power more gradually. at 26.5 v & 11 amp primary current I see splotchy coating develop on substrate. Also observe smoke in the chamber.

Now try the large (2") cylinder with heavy coated coils



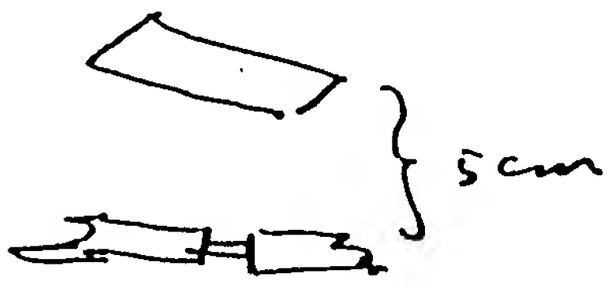
at 12.5 amp on filament started getting coating on substrate

Results - Nothing got thru the 1/4" hole in 2x2 glass to substrate.
2"x2" has some coating of blue & red.

Various spectra on charts.

80

Try Standard Condition to make beam 60 amps.



P_{He} = 380 on gauge

Today I Faxed to Wolfgang a copy of the spectrum of Ar which is a copy is shown on the attached page.

There appears the structure in the region between about 4500 Å and 7000 Å which is real. The curve of course shows why the material is reddish brown.

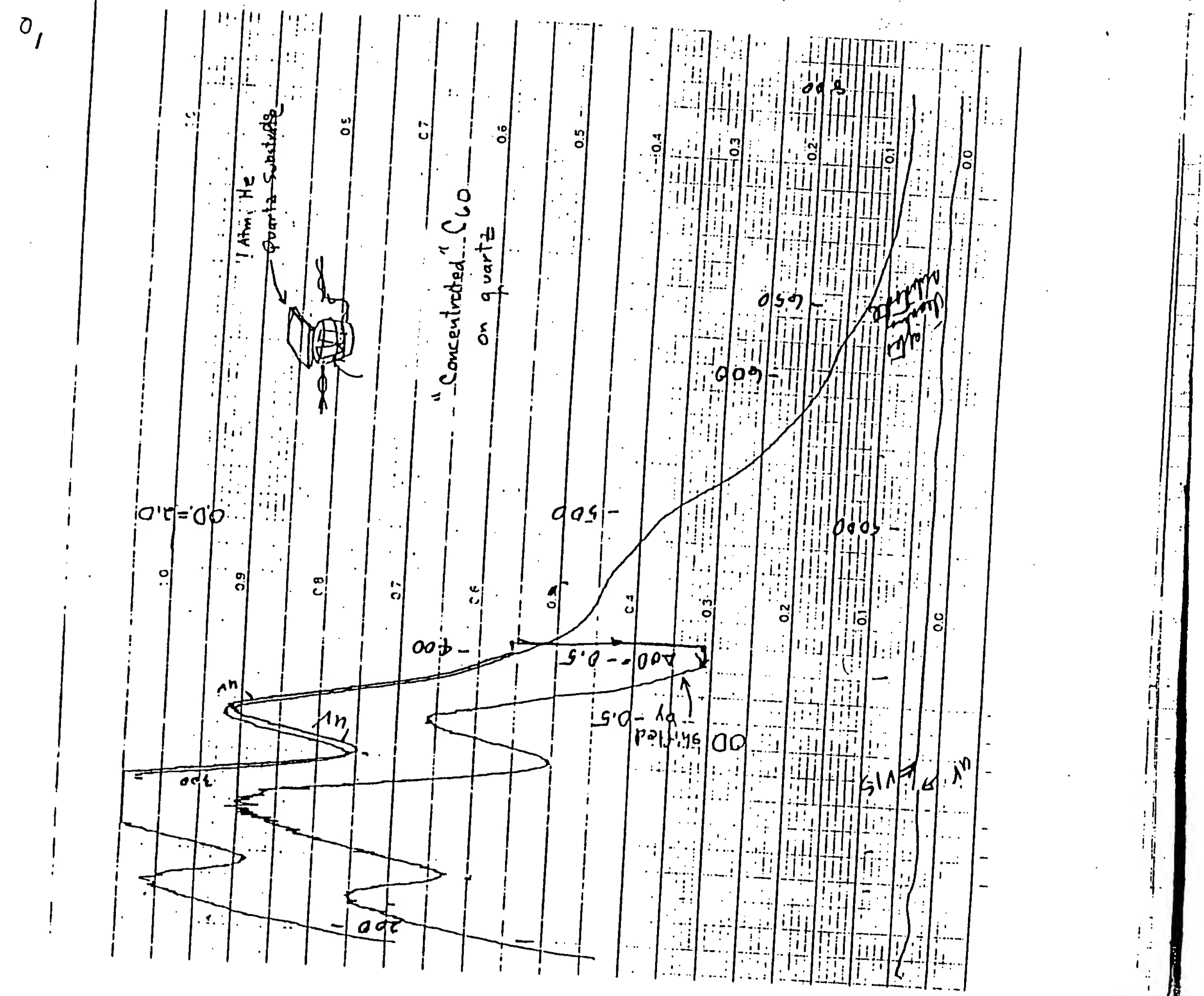
EXH. C-8

REDACTED

81

To: W. Kräutschmer

Spectrum of
"concentrated" smoke



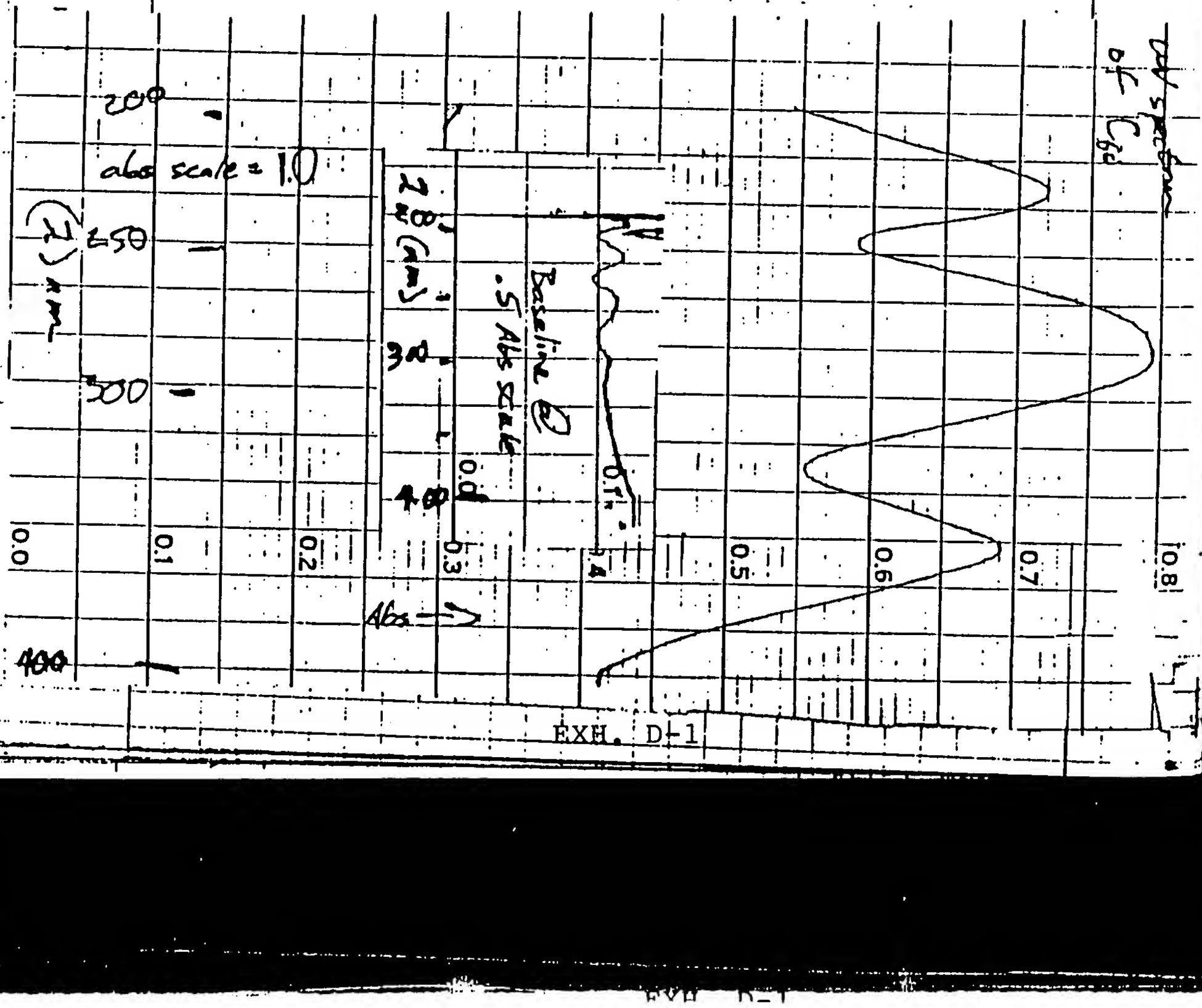
EXH. C-9

REDACTED

C_{60}

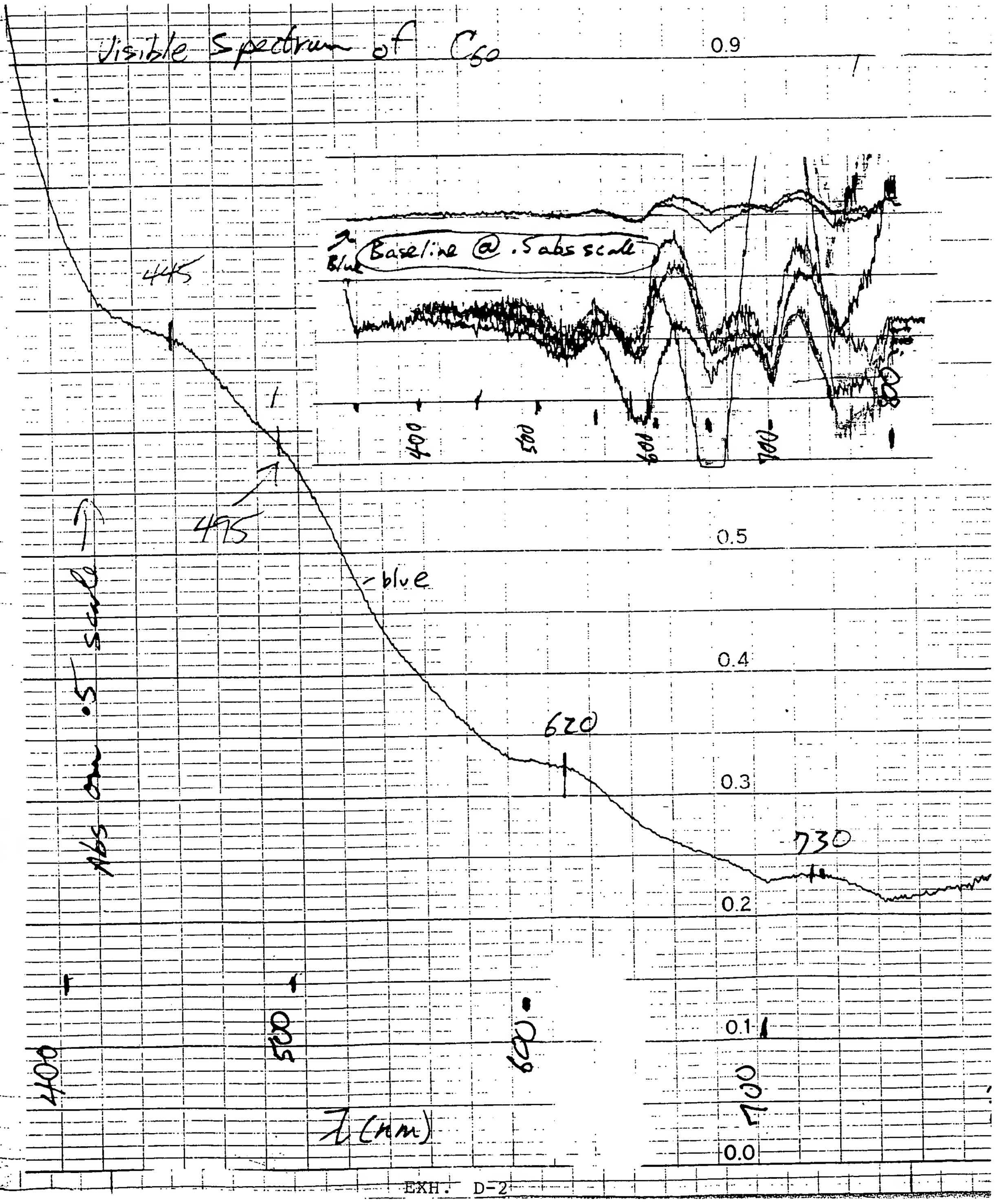
Today I made smoke & concentrated the C_{60} using sublimation
Taped in below is the UV scan of the sample,
and taped in on page 91 is the Visible scan.
There appear to be 4 absorption features superimposed
on the broad carbon feature in the visible. The 620nm
and 730nm may be instrumental error (see baseline).
The 445 and 495 are close to the 4428 A and
4882 nm DIB's!!!

Mr Clean



EXH. D-1

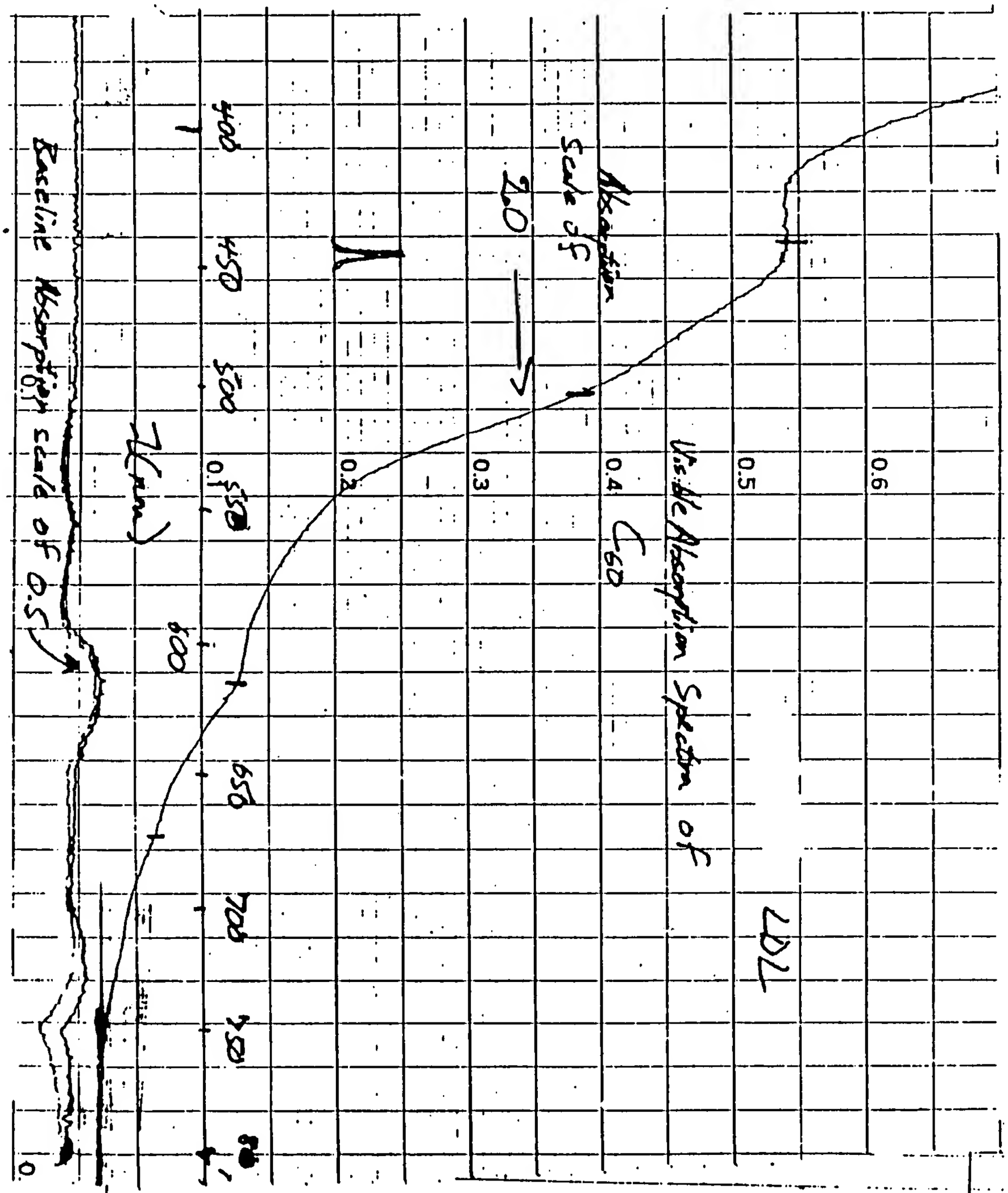
Visible Spectrum of C_{60}



EXH. D-2

REDACTED

Taped in below is a visible spectrum of C_{60}. I see features at 445, 500, 670 and possibly 620 nm. These may correspond to the 442 Å, 488 Å, 6660, and 6200 Å DIB.



EXH. E-1

~~Modification~~ Modifications to Smoke Production Method on pages 92 & 93.

Steps ① — Tip diameter ~ ¼"

Steps ② – Substrate-Crucible separation ~ 2 mm

Questions

① Why am I only seeing some of the DIBs? Specifically, why am I not seeing the 5800 Å feature?

Answer A

I am only seeing pure carbon60 and/or carbon60 with a trapped the features. The other DIBs are due to C_{60} with various ions trapped inside.

Answer B

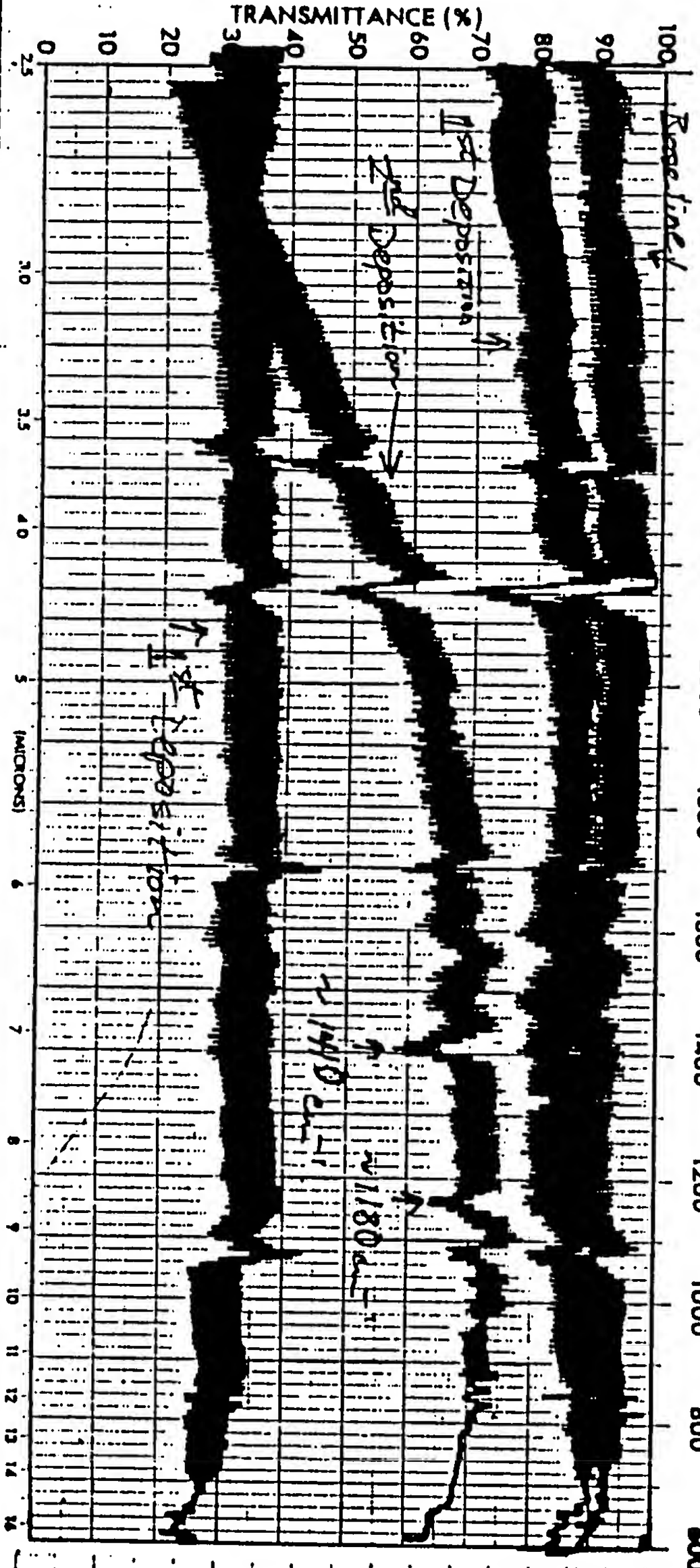
The other DIBs are due to C_{70}, C_{120}, ...

Taped in on page 98 is an IR scan of C_{60} on NaCl. Careful comparison of the baseline to the absorption spectrum shows only two features — at ≈ 1410 & 1180 cm⁻¹. These match up well to the Krätschmer et al features at ~~[redacted]~~ 429 and 1183 cm⁻¹. In his for this morning, he states that they are still seeing contamination due to C–H at 2900 cm⁻¹. I don't see that in my spectrum, so either it is not present or the instrument is not sensitive enough.

EXH. E-2

REDACTED

IR spectra of C_{60} on NaCl ✓



IR Scan of C_{60} on NaCl

SAMPLE 2

THICKNESS _____

DATE _____

OPERATOR _____

EXH E-3

[redacted]

REDACTED

Progress Report

[redacted]

EXH. F-1

[redacted]

REDACTED

EXH. F-2

REDACTED

IV UV-Visible

I have obtained good spectra of C_{60} from about 200-700 nm using the CARY 118. Scans are taped in on pages 90, 91, 96. I see visible features at 445, 500, 670, and possibly 620 nm.

V IR Spectra

An IR scan from 4000-800 cm^{-1} is taped in on page 98. I see the two features at 1429 and 1183 cm^{-1} and no other features. There seems to be relatively little contamination.

VI Near IR

Using the CARY 14, I've made some preliminary scans from 600-1600 nm. There is really only one candidate feature - at 1280 nm - but I don't have a baseline yet.

VII C_{60} Production

The most important thing I've done is refine our method for C_{60} production. I would estimate that we are now able to make it in ~ 0.1 gram batches. The purity appears to be very high.

EXH. F-4

EXH. F-5

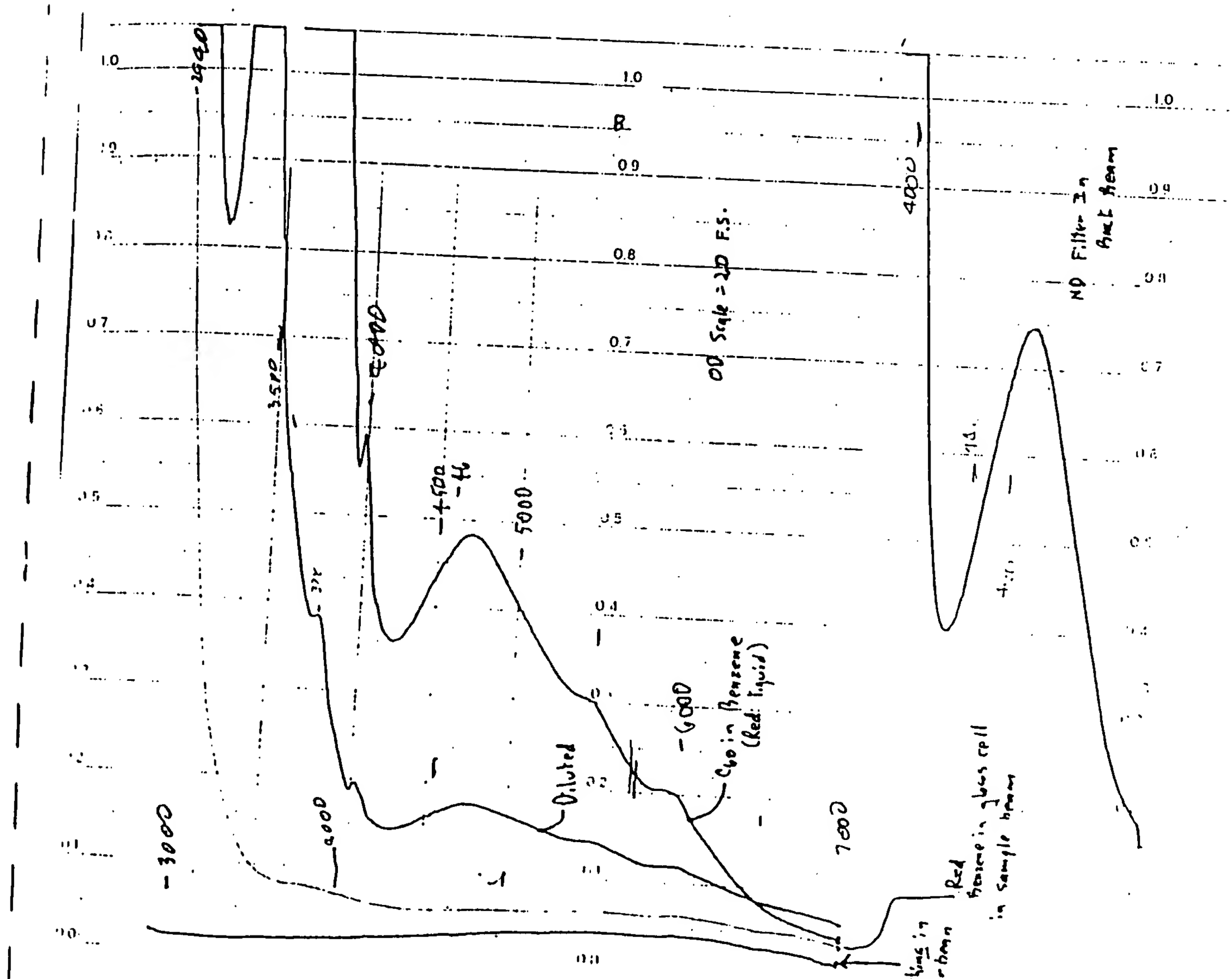
EXH. F-6

89

Ran a spectrum of C_{60} in benzene. ⇒ See attached page.

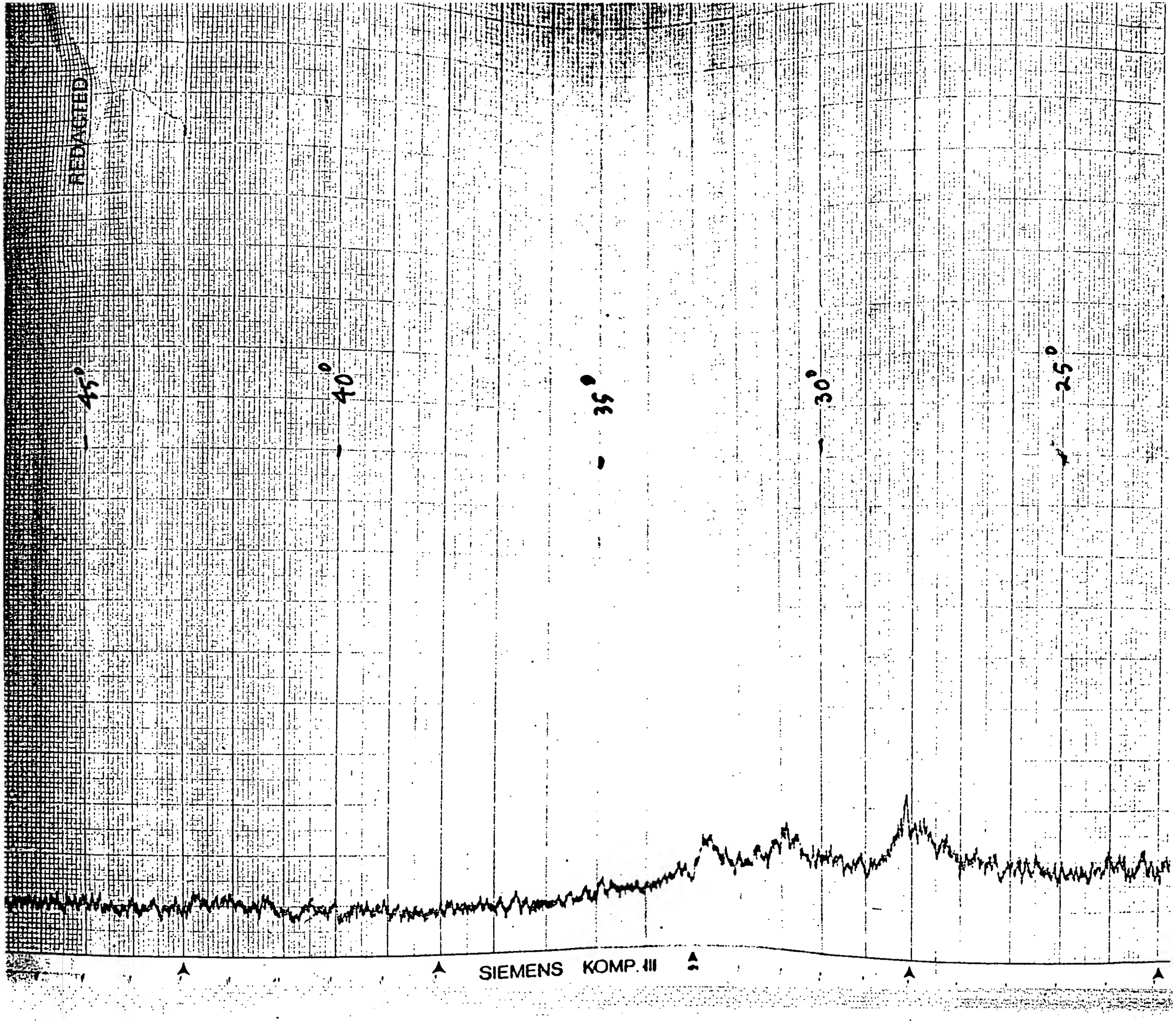
Saturday

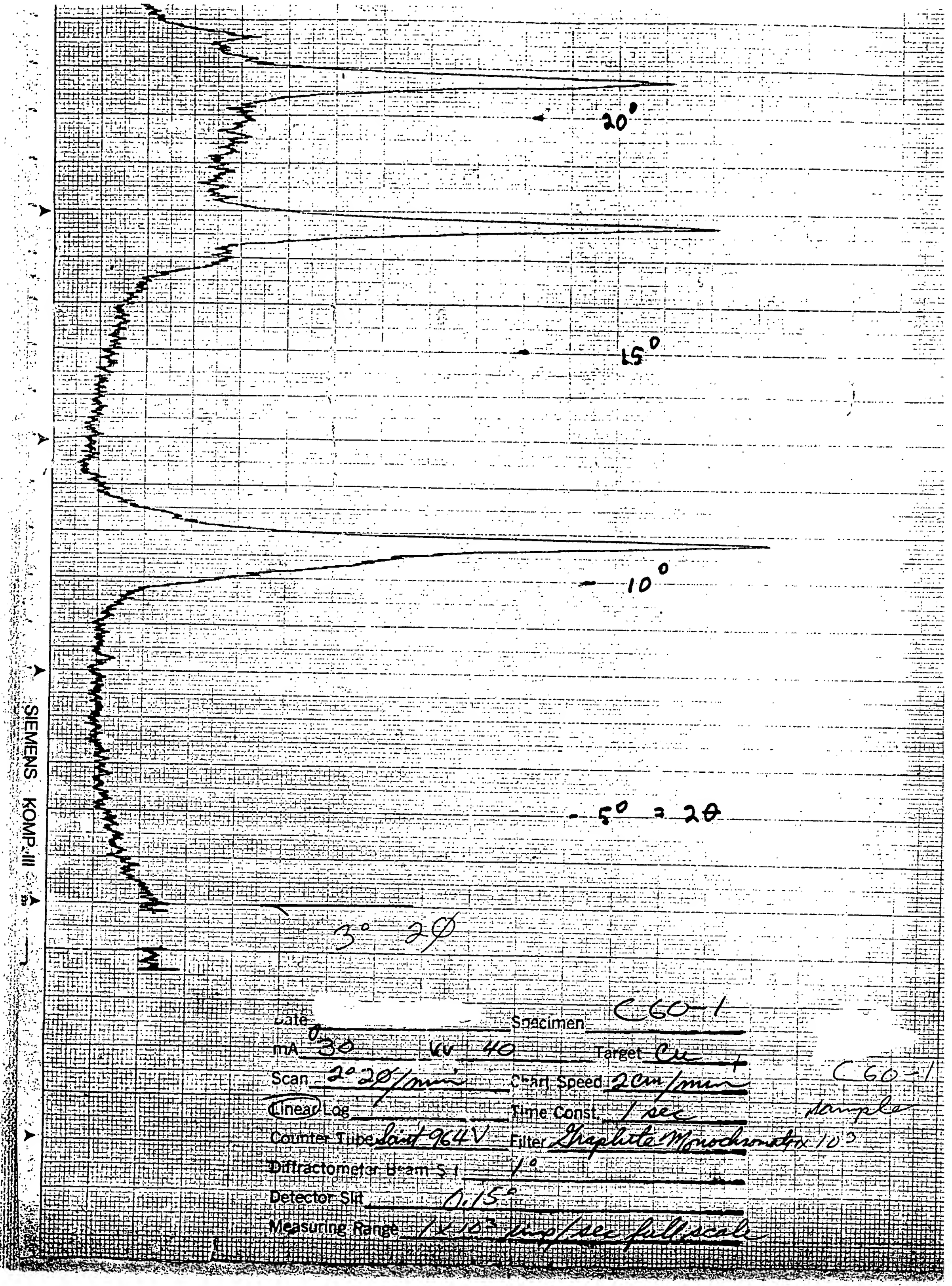
Came to lab about 3:00 P.M. Tried various solvents for C_{60}. Success was CS_2 and CCl_4 with a moderate apparent success for benzene. Failures included water, acetone, ethanol, methanol, propanol.



The above spectrum was measured on

EXH. G-2


REDACTED
45°
40°
35°
30°
25°
SIEMENS KOMP. III

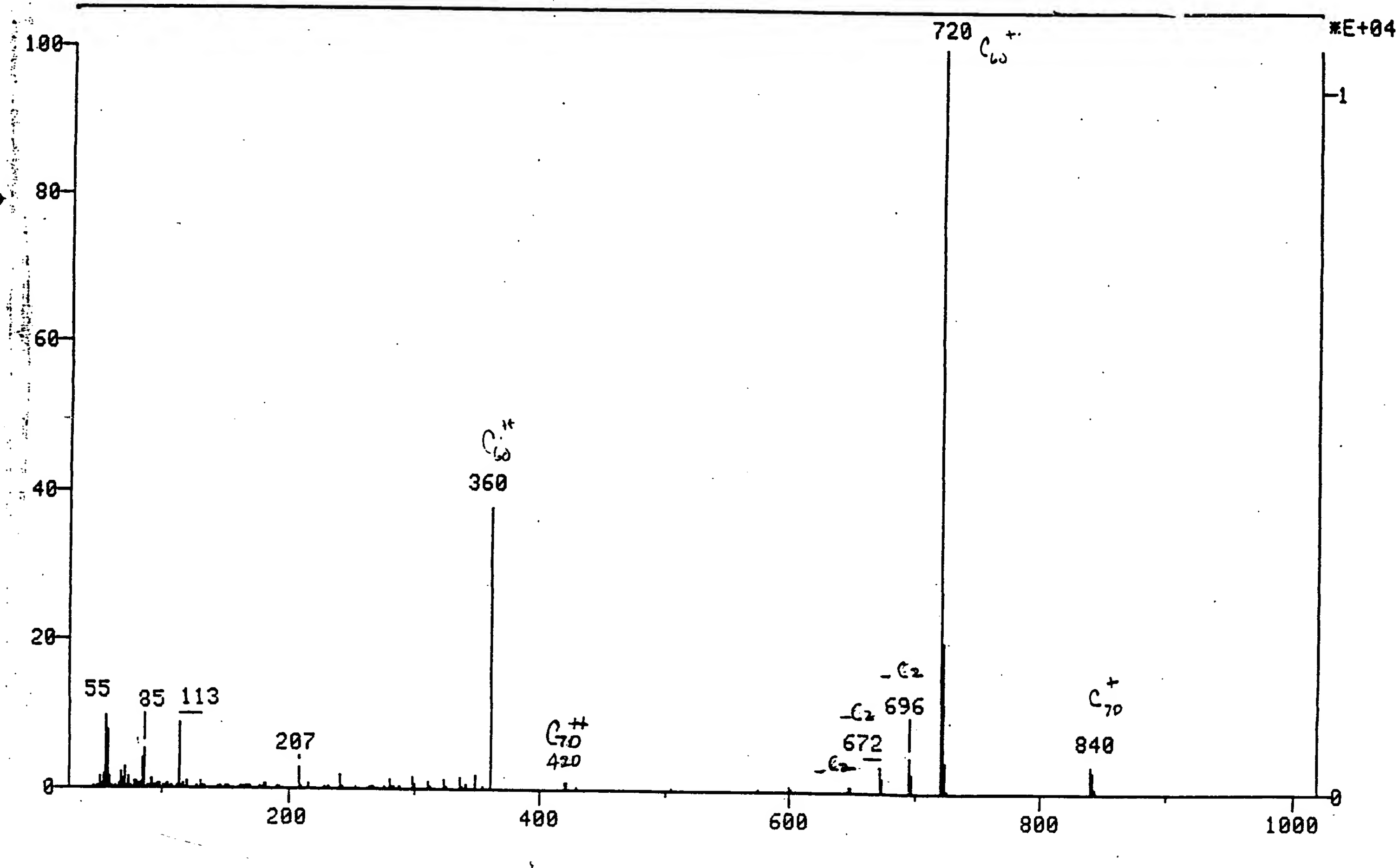

20°
15°
10°
5° = 2θ
3° 2θ
SIEMENS KOMP. III
Date
Specimen C60-1
mA 30 KV 40
Target Cu
Scan 2° 2θ/min
Chart Speed 2cm/min
Linear Log
Time Const. 1 sec
C60-1 sample
Counter Tube Xenit 964V
Filter Graphite Monochromator x 10³
Diffractometer Beam S 1 1°
Detector Slit 0.15°
Measuring Range 1 x 10³ imp/sec fullscale

EXH. H

SPEC: C60PURE ver 10 on UIC 2 2
Samp: PURE C60, TD=1.26, MRANGE=45-1000
Comm: HUFFMAN/LAMB/PHYSICS/422030
Mode: EI +VE +LMR BSCAN (EXP) UP LR
Oper: PFB
Base: 719.9 Inten : 10614
Norm: 719.9 RIC : 42018
Peak: 1000.00 mmu

Elapse: 00:16:07.0 472
Start : 13:01:05 633

Inlet : DIP
Masses: 45 > 1000
peaks: 211



EXH. I